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THE INTENSITIES OF THE K-SERIES X-RAY LINES OF TUNGSTEN AND PLATINUM

A DISSERTATION SUBMITTED TO THE
FACULTY OF THE DIVISION OF THE
PHYSICAL SCIENCES IN CANDIDACY
FOR THE DEGREE OF DOCTOR OF
PHILOSOPHY

DEPARTMENT OF PHYSICS
1939

By
WALDO H. KLIEVER

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A High Voltage, Demountable X-Ray Tube

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(Received January 23, 1939)

This tube consists of a minimum number of simple parts easily made in a university shop. It is designed for steady operation up to at least 110 kv, and permits convenient interchange of targets, replacement of filaments, etc.

TUBE BODY AND ENDS

THE tube is illustrated in Fig. 1. Its simplicity is aided by the use of Pyrex chemical piping,¹ as previously described by D. L. Webster *et al.*² A section 3×24 inches forms the body. It is normally supplied with flanged ends and a pair of metal flanges and asbestos inserts. The center was blown³ to 5 inches diameter to reduce wall thickness and to minimize effect of local charges on the glass causing wandering of the focal spot. This enlargement is not essential for most purposes.

End disks are 6 inches diameter of $\frac{3}{8}$ -inch sheet brass bolted against the glass with lead gaskets to form a vacuum-tight seal without need of any cement ($\frac{1}{2}$ -inch brass is preferable since the piping flanges have only three bolts). Spun brass hemispheres snap into grooves at the edge of the disks. Each end has a pumping outlet consisting of a standard 1-inch copper elbow, flattened where it is soldered into an elongated hole in the disks. The filament end also carries a one-turn copper cooling coil, soldered into a recess. Both end disks are nickel plated.

* With the Gaertner Scientific Corporation, Chicago, at time of this research. Now with U. S. Department of Agriculture, Leland, Mississippi.

¹ From Corning Glass Works, Corning, New York.

² D. Webster, W. Hanson and F. Duveneck, *Rev. Sci. Inst.* **3**, 729 (1932). Also, W. W. Hanson, *Rev. Sci. Inst.* **2**, 820 (1931); P. Kirkpatrick and P. Ross, *Rev. Sci. Inst.* **4**, 645 (1933); P. M. Harris, *Rev. Sci. Inst.* **8**, 478 (1937).

³ By General Electric X-Ray Corporation, Chicago.

GASKETS

The gaskets are cast seamless of pure lead. They fit round bottom grooves in the Pyrex piping and tube end disks, and have sharp rims which are easily pressed down to insure good contact. The mold consists of two cast iron disks, faced, and bolted together as shown in Fig. 2. Turned grooves form respective halves of the gasket. An additional concentric groove carries the molten lead to a port at the bottom of the gasket mold. It is closed by a plug which serves also to transmit a vent from the top of the mold.

The mold is smoked inside before each casting, and heated just to the melting point of lead before pouring. Gaskets are generally used only once, the bolts of the tube are drawn only moderately tight and usually no leaks are encountered at assembly.

FILAMENT ASSEMBLY

The filament assembly is supported by a $\frac{3}{8}$ -inch copper tube soldered to the end disk and to a $\frac{1}{4}$ -inch tube of Kovar metal⁴ connected by an adapter. A bead of Corning G705AJ hard glass is sealed to the end of the Kovar tube and to a $\frac{1}{8}$ -inch concentric Kovar rod carrying current to the filament. Near the filament this rod is supported on a mica washer.

The filament consists of 17 to 20 turns of 5-mil tungsten wire wound on a 1-mm mandrel

⁴ From Stupakoff Laboratories, 6627 Hamilton Ave., Pittsburgh, Pennsylvania.

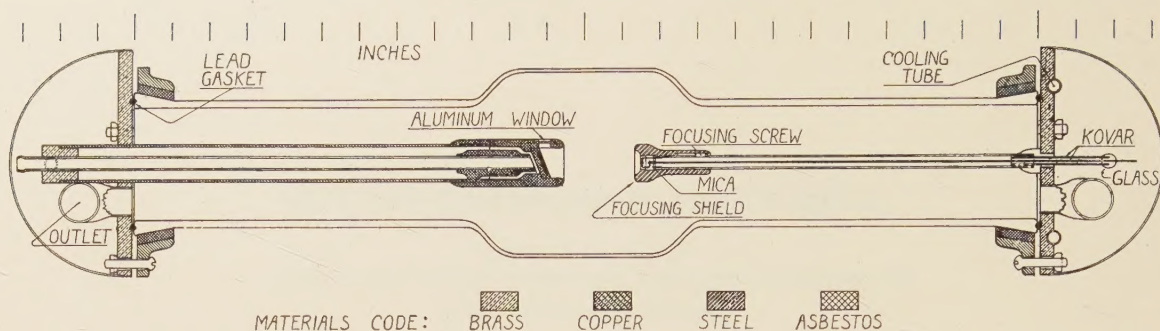


FIG. 1. High voltage x-ray tube of simple design.

with a copper wire spacer of similar dimensions. It is electric spot-welded to filament support posts of 20-gage nickel wire, silver-soldered to the copper tube and spot-welded to the Kovar rod, respectively.

The focusing shield is of steel, designed according to Beese,⁵ and slides on the copper tube. The focusing cup was milled by a $\frac{1}{4}$ -inch spherical end cutter moved $\frac{3}{8}$ inch linearly across the face. Focusing adjustment is by a hollow head set screw with ends flattened, lying with one half in a recess in the copper tube and the other half engaging a threaded groove in the focusing shield. A long-handled wrench permits adjustment through an opening (not illustrated) in the end of the tube to obviate disassembly.

TARGET ASSEMBLY

To prevent deflection of electrons to the glass envelope the target is placed in a recess or "well." The copper mount was turned to $1\frac{1}{4}$ -inch O.D. and to $\frac{13}{16}$ -inch I.D. except for a shoulder $\frac{1}{2}$ inch from the end. An inclined face on the shoulder at $22\frac{1}{2}^\circ$ was broached with a special punch driven with a hammer while the copper was hot. The punch consists of a steel tube with end milled to $22\frac{1}{2}^\circ$ and a central guide rod. A copper block about $\frac{3}{16}$ inch thick, carrying the target, is silver-soldered against the inclined face. Tungsten and platinum targets have been used. The copper block was cast about the tungsten in hydrogen atmosphere. The platinum is a 0.016-inch sheet, silver-soldered to copper. The aluminum window is 1 mm thick, securely held in a recess in the mounting by a copper frame and counter-sunk flat-head screws. This design insures good thermal conductivity from the window.

The target mount is soft-soldered to a copper support tube of 1 inch O.D. A $\frac{3}{8}$ -inch concentric copper tube carries cooling fluid. A filling body attached to its end insures a thin film of fluid (about $\frac{1}{16}$ inch thick) flowing at high velocity past the target and mount surfaces to promote quick change and remove bubbles.

The separation of target and filament assemblies is $1\frac{7}{8}$ inches.

OPERATION

The tube ends are supported by V-blocks of paraffin-impregnated wood. Kerosene, cooled by water, is pumped successively through the target assembly and filament end through glass leads

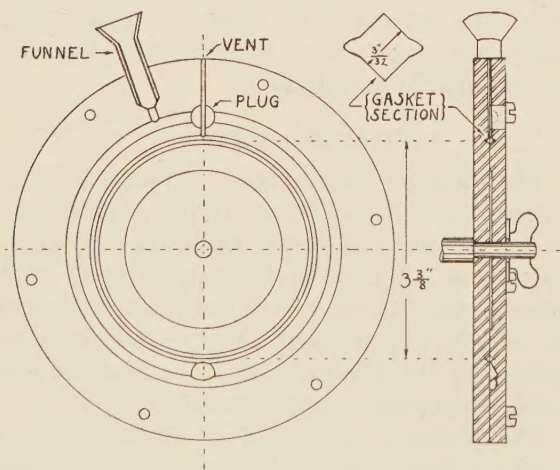


FIG. 2. Lead gasket mold.

⁵ N. C. Beese, Rev. Sci. Inst., 8, 258 (1937).

with short rubber tube connections. Glass pumping leads of 1 inch diameter are cemented with Picein to the outlets from which they converge to a liquid-air trap and ionization gage. Evacuation is by two three-stage mercury vapor pumps in parallel and a megavac forepump.

The tube is generally operated with both electrodes at d.c., filtered, high potential, equally above and below ground, from a transformer and two kenotrons.

Spark-over protection is provided by a nickel ring and concentric point placed in the pumping leads between tube and trap on each side, and connected to ground through heavy tungsten wires sealed in the glass walls. Further, to cushion accidental discharges, each end of the tube is connected to its respective filter condenser

through an inductance consisting of a single layer of No. 18 B. and S. enameled copper wire wound close on a $\frac{3}{4}$ -inch fiber tube for a distance of $38\frac{1}{2}$ inches. An additional 100,000-ohm protective resistance was found desirable when operating over 100 kv.

The tube has been operated continuously for periods as long as 11 hours at 110 kv and 3 ma. It has not been used at potentials above 120 kv or input power above 450 watts, but there is no indication that higher voltages and powers are not feasible.

Grateful acknowledgment is made to Dr. S. K. Allison, under whose direct supervision this research is being carried out, and to Messrs. M. L. Dannis and P. J. Zerwes for ideas and assistance.



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The Intensities of the K-Series X-Ray Lines of Tungsten and Platinum

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(Received June 1, 1939)

Measurements of the relative intensities of the $K\alpha$ and $K\beta$ lines of $_{74}\text{W}$ and $_{78}\text{Pt}$ have been made with a double crystal spectrometer, high pressure argon ionization chamber, and FP-54 Pliotron amplifier by measuring with a planimeter the areas under the curves of the lines graphed against Bragg angle. The results in the $_{78}\text{Pt}$ spectrum, which are the more reliable, are: on the basis 100 for the intensity of $K\alpha_1$: $K\alpha_2=52$, $K\beta_3=10.2$, $K\beta_1=20.0$, $K\beta_5=0.38$, $K\beta_2=7.8$, $K\beta_4=0.8$, $(O_{II}O_{III}-K)=1.2$.

Although the intensity of the quadrupole line $K\beta_5$ relative to $K\beta_1$ is in good agreement with the calculated value of Massey and Burhop, the predicted decrease in the relative intensity of this line at lower atomic numbers does not seem to be experimentally verified. The observed relative intensities of the stronger dipole lines seem to agree satisfactorily with the calculations of Pincherle and of Massey and Burhop.

MOST of the measurements of x-ray line shapes and intensities have been made only for elements of low atomic weights or were made photographically with x-ray spectrographs. This research was undertaken to study the K-series lines of heavy elements with a double-crystal spectrometer and ionization chamber, to look for anomalies and check against computed values for the intensities of regular and non-diagram lines.

I. THE TUBE AND SPECTROMETER

The x-ray tube, described previously,¹ was made at the Ryerson Laboratory, with metal ends, body of Pyrex piping, hot cathode and with target surrounded by metal shield with aluminum window. Electrodes were 48 mm apart. The measurements on the $K\alpha$ lines of tungsten were made with an earlier tube having similar

electrodes spaced about 37 mm apart and a glass envelope of 3" diameter throughout closed by large wax joints at the end. Tungsten target surfaces were tilted 30° with the perpendicular to the cathode beam; platinum, $22\frac{1}{2}^\circ$.

Power for the tube was supplied by a synchronous motor and 540-cycle generator, a transformer, 2 kenotrons and two 0.050-mf filter condensers in the usual voltage doubling circuit. A calibrated electrostatic voltmeter was connected between the target and ground and a 2- and 4-ma calibrated meter mounted at the tube indicated electron current. The filament current required repeated adjustments and both the voltage and current across the tube were continuously observed and maintained constant by rheostats at the observer's position near the spectrometer.

The double crystal x-ray spectrometer designed by S. K. Allison² was used with calcite crystals in (1, 1) position. The entrance slit to the ionization

* With the Gaertner Scientific Corporation, Chicago, at time of this research. Now with U. S. Department of Agriculture, Leland, Mississippi.

¹ W. Kliever, *Rev. Sci. Inst.* **10**, 126 (1939).

² S. K. Allison, *Phys. Rev.* **41**, 1 (1932).

chamber was 5 cm from the crystal B axis. Lead shields were placed about 1 mm from and perpendicular to the crystal faces to eliminate diffuse scattered radiation, especially that from crystal A . Both crystals were rotated, and thus a constant spot on the target was used as a source. The spectrometer arm carrying crystal B is geared to rotate at double the speed of crystal A . The ionization chamber slit was sufficiently wide so that the chamber did not need to be moved during related measurements except when comparing $K\beta$ lines with $K\alpha_1$.

One slit only was used to limit the rays, the other being set slightly wider. Since the collimating property of the two crystals serves to select a parallel bundle of rays, *viz.*, all those rays which have an angle of incidence equal to one-half the angle between the crystal faces, it follows that, using two slits of equal width, the full aperture of the slits is used only when the rays selected by the crystal enter exactly parallel with the line of the slits. Otherwise the beam will be limited by one side of one slit and the opposite side of the other slit, and the horizontal aperture will be less than the slit widths and will vary if there is an error in crystal rotation. Under conditions of 0.3 mm slits 138 mm apart the width would be reduced about $\frac{1}{4}$ percent for an accumulated error of only 1 second in the motion of crystal A .

The vertical aperture was 2.2° except during the earlier determinations on the tungsten $K\alpha$ lines when it was 1.1° .

The crystals were split calcite one of which was etched 3 sec. in dilute HCl according to the method developed at Cornell University.³ The full rocking curve width at half-maximum for parallel position is about $8\frac{1}{2}$ sec. of motion of crystal B only. A different pair of crystals used for the 74 W $K\alpha$ lines had similar resolving power for narrow slits but was found unsuited for use with the wide slits used on $K\beta$ lines.

II. IONIZATION CHAMBER AND AMPLIFIER

The ionization chamber was of steel of 25 mm diameter and 198 mm length internally, built for high pressure. The ends are bolted to flanges

which are screwed to the cylindrical body. The inner electrode is a 1.6 mm rod off center by 3.2 mm and insulated by amber, with surrounding grounded guard ring. The entrance window is 3.2 mm off center opposite the rod, covered by Celluloid with rubber gasket. The chamber body is insulated from the spectrometer, shielded by a 3.2-mm grounded lead housing, and charged to 90 volts above ground by an Edison storage battery. The ionization current was found satisfactorily constant from 30 to over 200 volts. Stability was improved by connecting a 4-mf condenser between chamber and ground and a 1-megohm resistor between chamber and battery. The chamber was built by T. M. Hahn and is generally similar in design to the steel chamber described by Williams.⁴

An FP-54 Plotron and L. and N. type R galvanometer were used in the well-known DuBridge and Brown circuit,⁵ modified according to Fig. 1f of Penick⁶ (modified Barth circuit). The tube was mounted in an evacuated, grounded metal case, with the additional precaution of a P_2O_5 drying tube. The ionization current conductor was supported only at the Plotron grid cap and at the chamber electrode, and was entirely contained in the vacuum. A key operated through a sylphon permitted grounding the grid or applying known voltages. The wires to the tube base were soldered to the prongs of the tube, the latter passing out of the vacuum through rubber gaskets. The entire circuit including lead wires was carefully shielded, and all batteries were contained in a double wall, heat insulated steel box.

The sensitivity of the circuit was 2×10^{-16} A/mm with the 8×10^{10} ohm grid resistor used, and deviations from linearity were found negligible.

III. CORRECTIONS TO READINGS

1. *Zero drifts* were determined by periodically closing a lead shutter at the spectrometer slits and taking zero readings which thus take account of such factors as stray radiation, variations in batteries, amplifiers, etc. The zero readings were

⁴ J. H. Williams, *Rev. Sci. Inst.* **3**, 586 (1932); T. M. Hahn, *Phys. Rev.* **46**, 149 (1934).

⁵ L. A. DuBridge and H. Brown, *Rev. Sci. Inst.* **4**, 532 (1933).

⁶ D. B. Penick, *Rev. Sci. Inst.* **6**, 116 (1935).

³ K. V. Manning, *Rev. Sci. Inst.* **5**, 316 (1934); F. K. Richtmyer, S. W. Barnes and K. V. Manning, *Phys. Rev.* **44**, 311 (1933).

plotted against the number of readings, and the interpolated value subtracted from each reading before plotting.

2. The *sensitivity* of the amplifier, when less than its maximum was determined by comparing the reading at some point on the background spectrum with that at full sensitivity, other conditions remaining equal. All results have been computed for 100 percent sensitivity by dividing the measured values by the sensitivity. It should be mentioned here as a possible help to others that it would have been advantageous had the sensitivity of the amplifiers been adjustable in steps instead of continuously, so that settings could be repeated and more thoroughly calibrated.

3. *Absorption* by materials between the target and ionization chamber requires some correction which has been applied only to the computed ratios of more intense lines. If a material of thickness x and density ρ absorbs 2 rays of wavelengths λ_1 and λ_2 and intensities I_1 and I_2 before and I_1' and I_2' after absorption, if the respective mass absorption coefficients are μ_1 and μ_2 , and if we let $I_1/I_2 = R$ (the true ratio) and $I_1'/I_2' = R'$ (measured ratio), then

$$R = \frac{I_1}{I_2} = \frac{I_1' e^{\mu_1 x}}{I_2' e^{\mu_2 x}} = R' e^{(\mu_1 - \mu_2)x}.$$

Only the absorption by the aluminum window and Pyrex wall of the tube was found appreciable. Absorption coefficients for aluminum were interpolated from curves plotted from data in the appendix of Compton and Allison's book on x-rays.⁷ From the same data the mass absorption

TABLE I. Sample data taken in an experimental run. Voltage on x-ray tube, 108 kv. Current through x-ray tube, 0.77 ma. Slit width, 0.06 cm. Sensitivity of amplifier, 75 percent of maximum.

X-RAY LINE	AREA (ARBITRARY UNITS)	AREA RATIO $K\alpha_2/K\alpha_1$	ATMOSPHERES PRESSURE IN CHAMBER	ABSORPTION CORRECTION IN PERCENT			FINAL INTENSITY RATIO
				AL	PY-REX	CHAMBER	
Pt $K\alpha_1$	492.7	0.507	83.1	—	0.4	—2.2	0.498
$K\alpha_2$	247.3			—	0.4	—2.2	
$K\alpha_1$	482.7			—	0.4	—2.2	
$K\alpha_2$	275.6	0.542	83.1	—	0.4	—2.2	0.532
$K\alpha_2(M)$	261.4			—	0.4	—2.2	

(M) indicates average of the two preceding determinations of area.

⁷ A. H. Compton and S. K. Allison, *X-Rays in Theory and Experiment* (Van Nostrand, 1935).

TABLE II. Intensity of $K\alpha_2$ on the scale $K\alpha_1 = 100$.

ELEMENT	EXPERIMENT	THEORY PINCHERLE	THEORY MASSEY AND BURROPS
⁷⁴ W	47	52	—
⁷⁸ Pt	52	—	—
⁷⁹ Au	—	—	48

coefficients for Pyrex were computed from an analysis supplied through the courtesy of Corning Glass Works with the following results: W $K\alpha_1$, 0.241; W $K\alpha_2$, 0.247; W $K\beta_1$, 0.214; W $K\beta_2$, 0.209; Pt $K\alpha_1$, 0.215; Pt $K\alpha_2$, 0.220; Pt $K\beta_1$, 0.195; Pt $K\beta_2$, 0.193.

No attempt is made to correct for absorption of rays in the target.

4. *Ionization chamber corrections.* Correction has been made with the aforementioned coefficient data for the unabsorbed rays. The maximum photoelectron range has been estimated as 0.2 cm for Pt $K\beta_2$ and 45 atmospheres (hardest ray and lowest pressure used) from the formula⁸ $x = v^2/b$ where v = photoelectron energy in electron kilovolts and $b = 610 \times 45$. Thus the photoelectrons should be completely absorbed. Fluorescent radiation is assumed largely used in the Auger effect. Scattered radiation, modified and unmodified, has been neglected in its effect on line intensity ratios, because no convenient methods of correcting for it were found.

Considerable diffuse scattering by the calcite crystals was observed, which necessitated careful shielding. This may have affected the intensity ratios, but no corrections were attempted for this factor.

IV. RESULTS

Table I shows a sample of the data taken in an experimental run. The intensities are taken (before correction) as proportional to the areas under the curves with Bragg angle as abscissae, as measured with an Ott planimeter.

The $K\alpha$ line intensities

Considerable interest is attached to measurements of the intensities in the K series of heavy elements, because of the chance to detect relativistic effects, if present. The nonrelativistic

⁸ Reference 7, page 494.

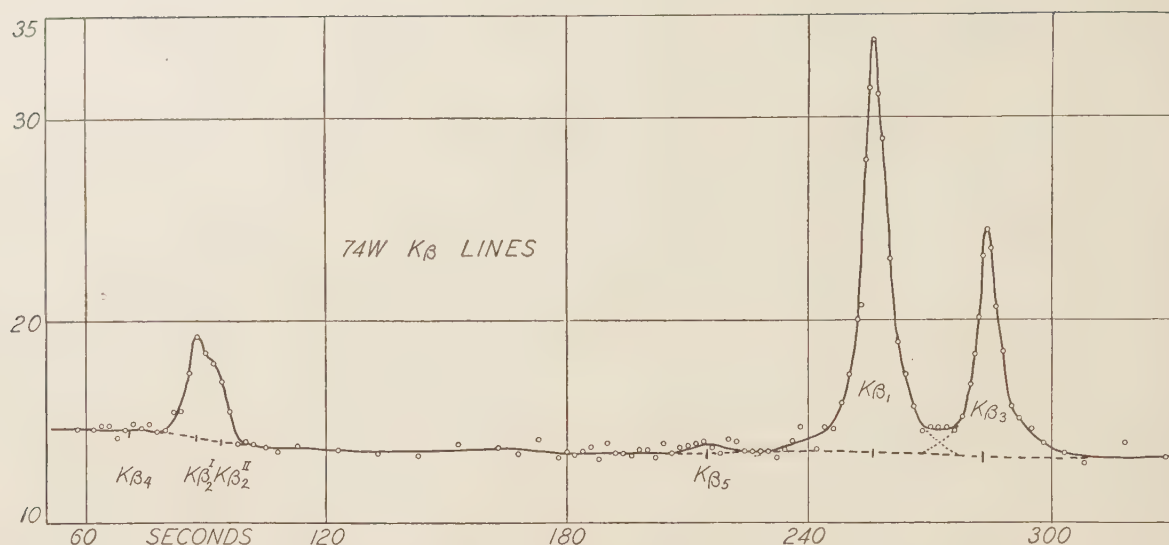


FIG. 1. Intensity curve of ^{74}W $K\beta$ lines. The short cross lines on Figs. 1 and 2 give the positions of the x-ray lines according to Ingelstam, as measured from $K\beta_2^I$ and $K\beta_3$.

wave functions are certainly inadequate for the representation of these states in which the K electrons are bound with energies of approximately 70 kev. Massey and Burhop⁹ have made calculations of x-ray intensities in which a rough approximation to a rigorous relativistic treatment has been introduced, and similar calculations have been made by L. Pincherle.¹⁰ Massey and Burhop state that the relativistic treatment does not alter appreciably the nonrelativistic value of 0.500 for $K\alpha_2/K\alpha_1$, even in ^{92}U . A previous experimental value of 0.500 was found by Duane and Stenström¹¹ in ^{74}W . Table II summarizes the results on the $K\alpha$ lines.

No definite evidence of $K\alpha_3$ (L_I-K) was obtained, although some of the curves taken on tungsten showed slight irregularities in the expected region. According to Massey and Burhop, this line should be about as strong as $K\beta_5$ in ^{79}Au .

The $K\beta$ line intensities

Since the advent of high resolving power spectrometers for x-rays, the β -group in the K series has been reexamined and several new, faint lines reported.¹² Since this work was begun, an

excellent monograph by Ingelstam¹³ has appeared, in which accurate measurements of the wave-lengths of the K lines of the heavy elements have been made with a high resolving power photographic spectrograph. Some visual estimates of intensities are made in this work.

Figures 1 and 2 show the present results on the $K\beta$ lines of ^{74}W and ^{78}Pt . Comparison of the curves shown on the $K\beta_2$ ($N_{II}N_{III}-K$) doublet with Fig. 23a of Ingelstam's monograph, and Fig. 4 of Richtmyer and Barnes¹⁴ indicates that the resolving power was comparable to, but slightly lower than, that of the two other researches.

TABLE III. Intensities of the $K\beta$ lines of ^{74}W and ^{78}Pt , based on the scale $K\alpha_1=100$.

SYMBOL TRANSITION	^{74}W λ IN X.U.	^{74}W OB- SERVED INTEN- SITY	^{78}Pt λ IN X.U.	^{78}Pt OB- SERVED INTEN- SITY	THEORETICAL INTENSITY	
	INGEL- STAM		INGEL- STAM		CHERLE 74W	MASSEY 79Au
$K\beta_3$ $M_{II}-K$	184.795	9.6	164.157	10.2	11	13
$K\beta_1$ $M_{III}-K$	183.991	18.1	163.334	20.0	21	29
$K\beta_2^{II}$ $M_{IV}-K$	182.882	0.25	162.364	0.38	—	0.19
$K\beta_2^I$ M_V-K	182.711		162.203		—	0.41
$K\beta_2^{II}$ $N_{II}-K$	179.232	1.7	159.053	3.0	2.5	—
$K\beta_2^I$ $N_{III}-K$	179.049	3.1	158.863	4.8	4.5	—
$K\beta_4$ $N_{IV}N_V-K$	178.55	0.036	158.48	0.8	—	—
— $O_{II}O_{III}-K$	178.073	0.04	157.922	1.2	1.5	—

⁹ H. S. W. Massey and E. H. S. Burhop, Proc. Camb. Phil. Soc. **32**, part 3, p. 461 (1936).

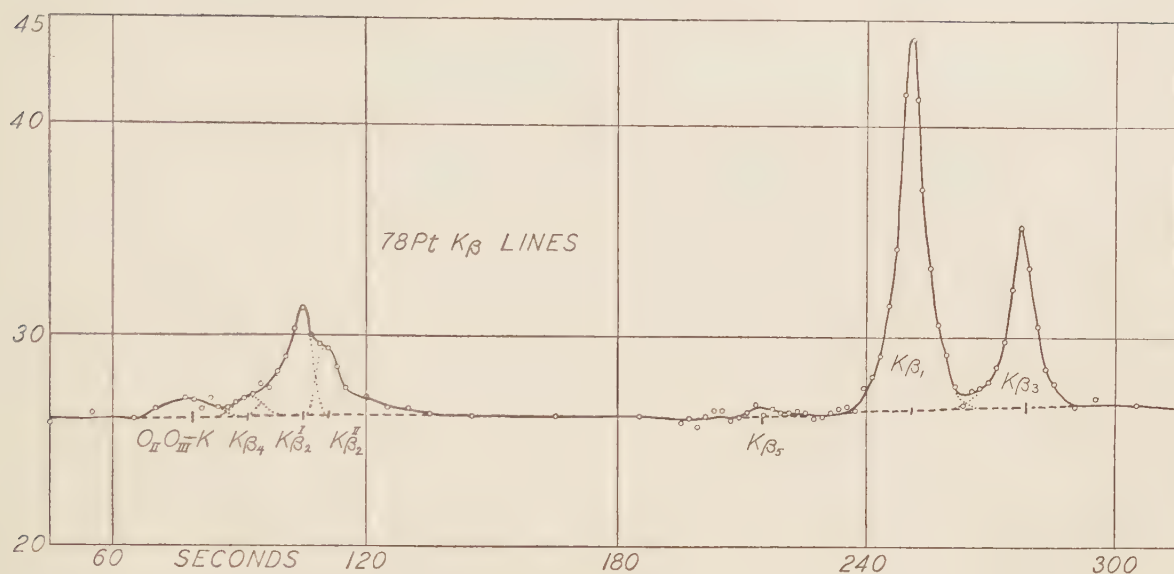
¹⁰ L. Pincherle, N. Cimento **10**, 344 (1933).

¹¹ W. Duane and W. Stenström, Proc. Nat. Acad. Sci. **6**, 477 (1920).

¹² A. H. Compton and S. K. Allison, *X-rays in Theory and Experiment* (Van Nostrand, 1935), pp. 632, 639.

¹³ E. Ingelstam, Die K-Spektren der Schwere Elemente, Nova Acta Regiae Societatis Scientiarum Upsaliensis, Ser. IV, **10**, No. 5 (1937).

¹⁴ F. K. Richtmyer and S. W. Barnes, Phys. Rev. **46**, 352 (1934).


 FIG. 2. Intensity curve of ^{78}Pt $K\beta$ lines.

In addition to the diagram lines $K\beta_1$, $K\beta_3$ (M_{II} , $M_{\text{III}}-K$) and $K\beta_2^{\text{I}}$, $K\beta_2^{\text{II}}$ (N_{II} , $N_{\text{III}}-K$) which were known previous to Ingelstam's work, he reports evidence of $O_{\text{II}}O_{\text{III}}-K$, obtained first in ^{49}In and observed in heavier elements also. In the ^{78}Pt $K\beta$ spectrum given here, there is some evidence of a broad line in the required region. Ingelstam gives 157.922 x.u. as the wave-length; the maximum of the broad peak in the present work lies at 158.05 x.u. In the present work on tungsten, the corresponding line was not detected with certainty. Table III gives the results on the $K\beta$ lines.

The intensities given for the nondiagram lines $K\beta_5$ and $K\beta_4$ certainly have a large error, probably a factor of 2, because they lie so near the base-line. In the case of tungsten, the values seem less reliable than for platinum. The platinum curves were taken subsequent to the tungsten experiments, when the technique had been better developed.

If one takes the observed values of the intensity of $\text{Pt } K\beta_5$ with respect to $\text{Pt } K\beta_1$, namely 0.019, it agrees well with Massey and Burhop's value of 0.021 for this ratio. Although the intensity of this interesting quadrupole line agrees with Massey and Burhop's calculation at ^{78}Pt , the meager experimental evidence available would seem to show that its intensity does not decrease with atomic number as they predict. At ^{44}Ru , Wilhelmy¹⁵ estimated $K\beta_5$ as 0.25 if $K\alpha_1$ is 100, whereas for ^{51}Sb according to Massey and Burhop's calculations, the line should have sunk to $\frac{1}{5}$ of its value at ^{78}Pt , which means a value of 0.05–0.08 instead of Wilhelmy's 0.25.

The author wishes to express his thanks to Dr. S. K. Allison, under whose guidance the work was done, and to M. L. Dannis for assistance and for measuring the areas of the curves.

¹⁵ E. Wilhelmy, Phys. Rev. **46**, 130 (1934).

